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STRESS CORROSION CRACKING OF WROUGHT AND P/M HIGH  
STRENGTH ALUMINUM ALLOY (U) CARNEGIE MELLON UNIV  
PITTSBURGH PA DEPT OF METALLURGICAL ENGT

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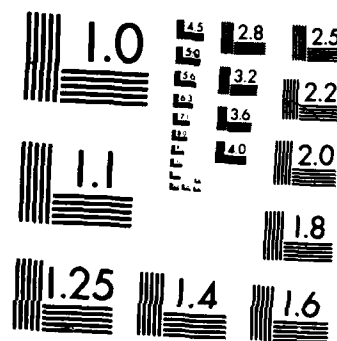
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The major findings from a comprehensive study on the role of microstructure on the susceptibility to environmental embrittlement of high strength aluminum alloys are presented and discussed. Most of the studies used commercial 7075 or a high purity equiaxed version, HP7075, or a similar powder version 7090. Through the innovative use of loading mode and straining electrode tests, stress corrosion cracking was shown to be controlled by the introduction and internal distribution of hydrogen, particularly to grain boundaries. This was the case for the underaged and peak aged microstructures, with the latter being the most susceptible. The SCC behavior of the even more resistant

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STRESS CORROSION CRACKING OF WROUGHT  
AND P/M HIGH-STRENGTH  
ALUMINUM ALLOYS

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FINAL REPORT

Air Force Office of Scientific Research

Grant AFOSR-81-0041  
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ABSTRACT:

The major findings from a comprehensive study on the role of microstructure on the susceptibility to environmental embrittlement of high strength aluminum alloys are presented and discussed. Most of the studies used commercial 7075, or a high purity equiaxed version, HP7075, or a similar powder version 7090. Through the innovative use of loading mode and straining electrode tests, stress corrosion cracking was shown to be controlled by the introduction and internal distribution of hydrogen, particularly to grain boundaries. This was the case for the underaged and peak aged microstructures, with the latter being the most susceptible. The SCC behavior of the even more resistant overaged microstructure was shown to be controlled by anodic dissolution processes, predominantly associated with slip bands. Aluminum alloys were also shown to be susceptible to embrittlement under conditions of cathodic polarization correcting a widely held belief that concomitant anodic processes are required. Studies using controlled microstructures were able to rank the importance of different microstructural features to help develop alloy design strategies for more environmentally resistant alloys --the most effective features are those grain interior precipitates which promote fine, homogeneous slip either by reduced particle cutting or enhanced dislocation generation; fine, grain boundary precipitates which can strongly but innocuously trap hydrogen were the next most effective feature; precipitate free zones were found not to be very important, except in their role in reducing the local strength of the boundary region. Mechanistically, hydrogen transport by mobile dislocations could dominate the embrittlement process, which itself results from either highly localized slip, lattice softening or apparently in some cases grain boundary hydride formation.

Overview: The goals of this scientific report are to first briefly summarize the major results for the contract/grant covering the period prior to 1981, to use those results to reiterate the goals of the just concluded five year grant, to detail the major findings of this research, and to attempt to arrive at general conclusions regarding stress corrosion cracking (SCC) and hydrogen embrittlement (HE) of high strength aluminum alloys. In particular, we will set guidelines and a framework for alloy design to optimize resistance to environmental resistance, as well as suggesting possible mechanisms for SCC and HE.

Major Results From Previous Grant Period: Research initially focussed on wrought 7075 and a high purity version of the alloy where it was possible to obtain large equiaxed grains; use of these two versions enabled a separation of the contravening effects of grain shape and associated microstructural features to be made. The following is a summary of the most important findings from this period:

1. It was unequivocally shown that under conditions of cathodic polarization that the introduction of hydrogen could lead to measurable and reproducible embrittlement of 7075, as manifested by a reduced reduction of area in a tensile test (1).\*
2. A temperature dependent loss of ductility was shown for a broad range of microstructures ranging from underaged to peak aged

\*Cited references are peer reviewed papers from this research program.

to overaged, with the loss being in general highest for the underaged and lowest for the overaged. It was also shown that a maximum in loss was found at intermediate strain rates and an increase in degree of temper decreased the sensitivity at the strain rate of maximum effect(2,3).

Similar behavior patterns were found for HP7075 and two other aluminum alloys 7050 and 2124, demonstrating the generality of the hydrogen effect (4,5).

3. The next major accomplishment was the demonstration that hydrogen can also play a major role in the stress corrosion cracking of high strength aluminum alloys in salt-water like environments, conditions that have invariably been ascribed to anodic dissolution control(6). The approach taken was to extend preliminary studies of the use of loading mode to discriminate between SCC and HE. In brief, differences in failure times or cracking behavior should be expected between Mode I(tensile) and Mode III(torsional) loading if hydrogen is an active and likely controlling participant in environmental embrittlement, while a much smaller sensitivity should be observed if anodic dissolution controls. The former was found to be the case, as illustrated in Figure 1, which summarizes the response of short transverse 7075 T6 to loading mode in 3 1/2 % NaCl solution. The large differences in the normalized fracture stress intensities verify the importance of hydrogen, a point we will return to when we discuss loading mode results in other materials, as well as mechanistic implications of the observed behavior (7,8).

Major Results From Current Grant Period: The ensuing discussions will center in three main areas: 1). An extension of the detailed characteriz-

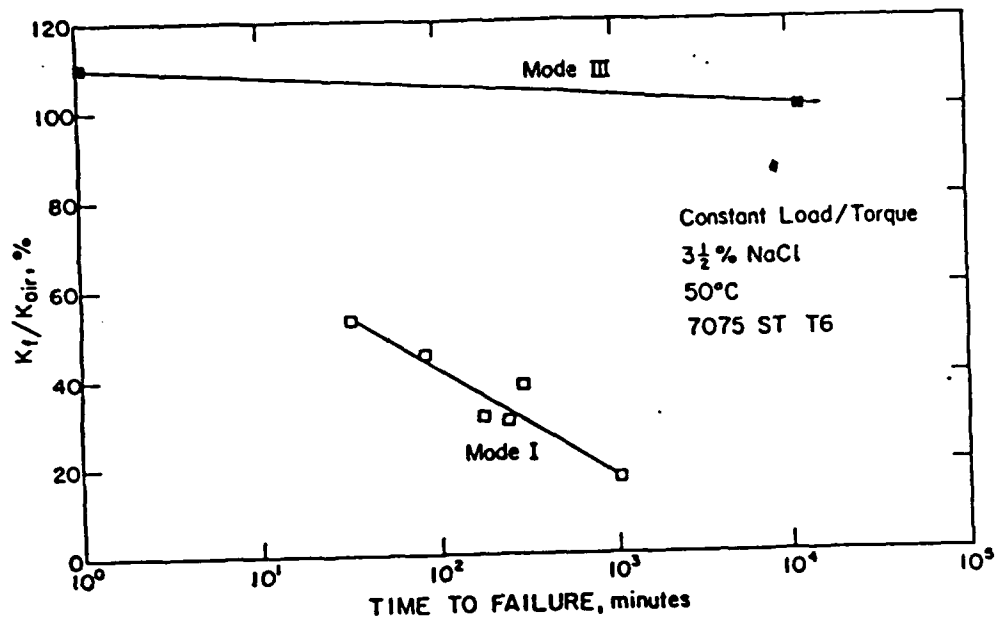


FIGURE 1. LOADING MODE RESULTS FOR 7075 ST-T6

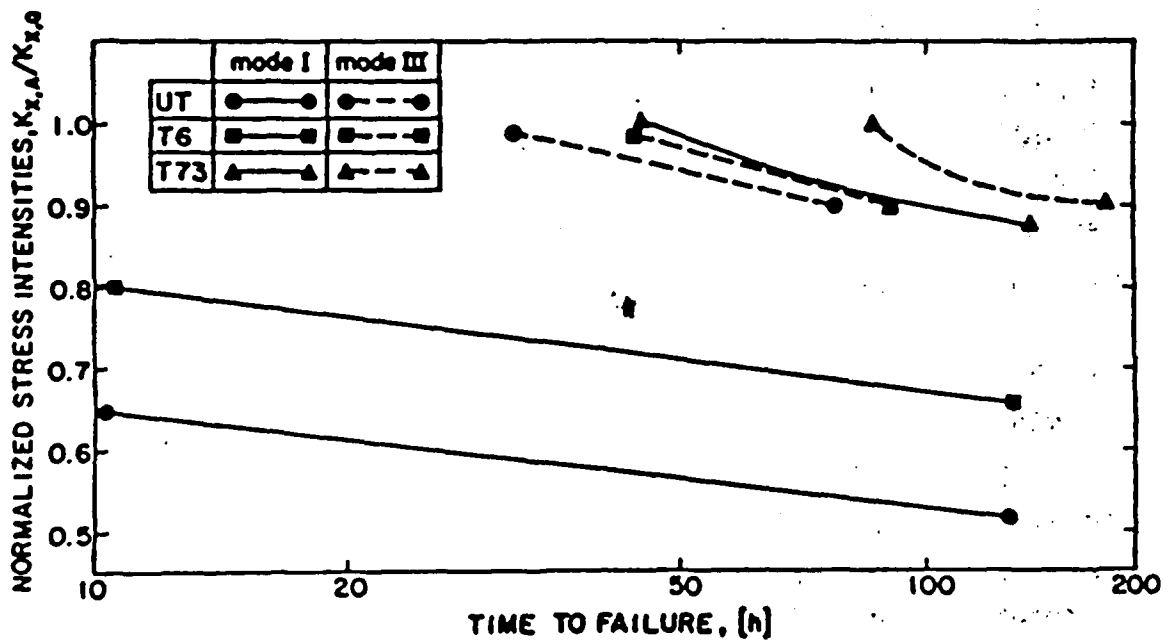


FIGURE 2. SUMMARY OF SCC RESULTS IN 1N ALUMINUM CHLORIDE FOR 7075



ation of wrought 7075 to other wrought alloys and importantly to the powder based alloy 7090; 2). Suggestions for identifying responsible mechanism(s) for SCC and HE; 3). Development of alloy design guidelines to optimize existing and to develop new aluminum alloys more resistant to environmental embrittlement and hopefully without a concomitant penalty in strength and/or toughness.

1). The 7075 studies were extended to examine both different tempering conditions and different environmental solutions. For the former, the effect of a new type of heat treatment, retrogression and reaging, on environmental susceptibility was studied (9).

This treatment which involves a partial resolutionizing followed by a step anneal and reage after quenching, was reported to provide a T6 strength with the environmental resistance of a T73 temper. Our studies incorporating loading mode tests in 3 1/2% NaCl at 50C and tensile testing with concomitant cathodic charging (SET test) in pH1 HCl, did not support this contention(9). SCC resistance of notched specimens with the T6-RR temper was found to be no better than the conventional T6 temper. It was pointed out that while this insensitivity could reflect differences in the relative contributions of crack initiation and propagation, or fact that our RR heat treatment was not the optimum one, the susceptibility of the RR temper to hydrogen suggests that this heat treatment be used cautiously for applications where hydrogen is present or could be generated.

SCC studies were carried out in 7075 in a 1N HCl solution of  $AlCl_3$  as a function of temper and loading mode(10). This solution has been used in a number of laboratories for accelerated testing and we believed it important to establish whether this solution provided an appropriate test environment to mirror SCC behavior in other solutions. It also provided an

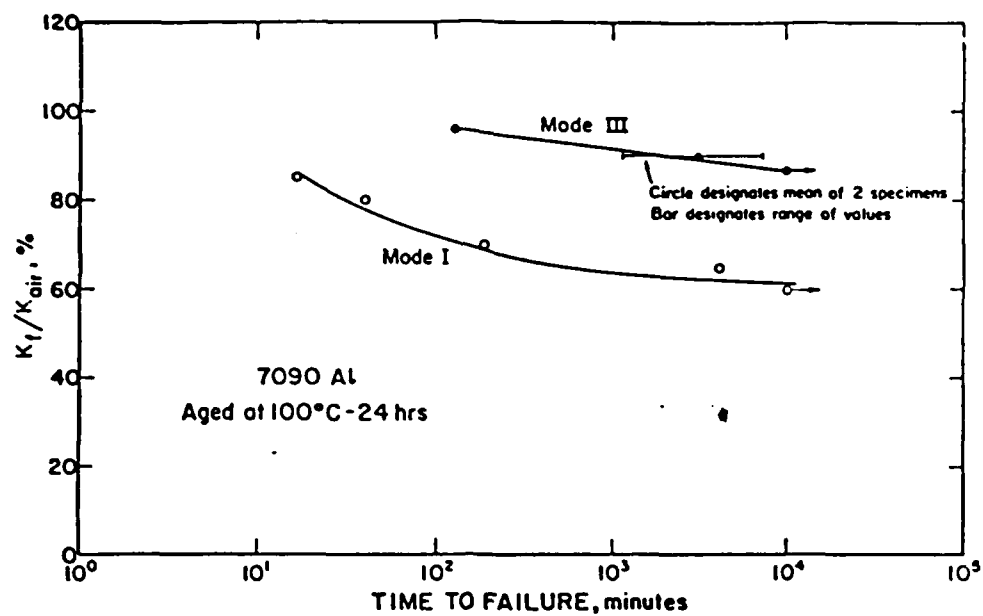


FIGURE 3. LOADING MODE RESULTS FOR 7090 UT

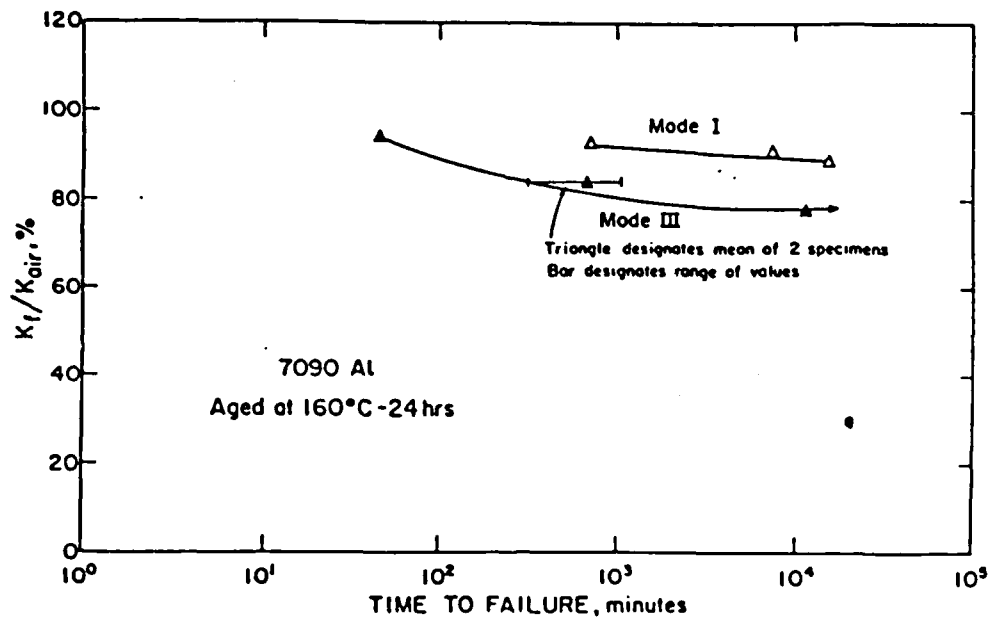


FIGURE 4. LOADING MODE RESULTS FOR 7090 OT

environment where problems of concurrent generalized corrosion must be taken into account. It was found that in the most widely used temper for this alloy, T73, that a highly aggressive environment like  $\text{AlCl}_3$  can add a strong non-SCC component to cracking and therefore lead to possible misinterpretations of accelerated laboratory SCC tests conducted in this and like environments.

These studies also confirmed that, as in salt water, 7075 SCC resistance is sensitive to both loading mode, see Figure 2, demonstrating the importance of hydrogen, and to temper, with the UT being most susceptible and T73 the least. From detailed fractographic studies it was also established that HE is clearly the predominant mechanism for materials in the UT temper, while anodic dissolution controls for the T73 temper, consistent with the greater corrosion rate for the latter. A specific contribution of different microstructural features was also identified and we will return to this when we discuss mechanism and alloy design strategies.

2) As part of our interest in establishing the generality of such findings, a detailed SCC/HE study was undertaken with the powder based high strength alloy, 7090. Although this alloy is similar to 7075, the decreased Zn concentration and the presence of Co affects the aging sequence and the powder processing does effect the final grain structure. Either or both of these differences could influence SCC behavior. Significant differences in micro and macrostructure were in fact found. The 7075 alloy had a recrystallized disc-like grain structure,  $400\mu\text{m}$  in the longitudinal direction,  $100\text{--}200\mu\text{m}$  in the long transverse and  $50\mu\text{m}$  in the short transverse, while the 7090 exhibited a partially recrystallized microstructure with fine, nearly equiaxed grains approximately  $2\mu\text{m}$  in the longitudinal direction,  $1\mu\text{m}$  in the long transverse and  $1\mu\text{m}$  in the short transverse direction.

As expected, 7090 also contained a fine dispersion (0.01-0.04 $\mu$ m) of oxide particle stringers aligned in the extrusion direction and located mainly at grain boundaries, supporting their role in inhibiting grain growth. The major microstructural differences, compared to 7075, was the relative absence of T-phase and the presence of 0.1-0.4 $\mu$ m spherical Co rich particles at grain boundaries. Smaller differences between the two were found in PFZ width and the size and location distribution of  $\eta$  and  $\eta'$  particles for similar aging sequences.

Largely because of the finer grain size, 7090 was considerably stronger than 7075 for similar agings, although the tensile ductility and fracture stress intensities for notched tensiles were quite similar. SCC testing was carried out in a brine solution to which chromate was added to reduce the severe exfoliation damage found, particularly in the overaged temper; both the effects of loading mode and crack initiation and propagation of bolt loaded DCB specimens were monitored in this solution. SET testing was carried out in pH1 HCl at room temperature. Significant similarities and differences were observed in the SCC/HE behavior of 7090 compared to 7075 (7).

In the underaged and peak aged conditions, 7090 showed a sensitivity to loading mode similar to 7075, suggesting a strong contribution of hydrogen control to stress corrosion cracking in general for these two tempers; this is illustrated in Figure 3. The overaged temper is much more resistant to environmental embrittlement likely through an enhanced resistance to crack initiation. SCC lifetime is found to be now shortest in mode III, Fig.4; this may be related to differences in crack opening and an enhanced local dissolution rate for the more closed crevice in mode III loading. This strong role of anodic dissolution in SCC of overaged structures was also found in 7075 tested in AlCl(3) and may well be a general characteristic of this condition.

3) Throughout the course of this program a major if not the main thrust has been to provide evidence to support the thesis that microstructural control of SCC and HE is not only feasible but is desirable, inasmuch as it is the dominant variable for both hydrogen and anodic dissolution controlled processes. Support for this approach came early with our findings that different microstructures with similar strength levels could display orders of magnitude differences in environmental response and that this effect persisted for both loading modes and for different solution chemistries and polarization conditions. It was important to extend such observations to another level of detail to discriminate insofar as possible the specific roles of grain size and structure, precipitation free zones, constituent particles, and grain boundary and grain interior strengthening precipitates. With respect to grain structure, we have shown in 7075, in agreement with others, that short transverse SCC properties are greatly inferior to longitudinal. This is illustrated in Figure 5, which compares the UT temper for both loading modes. Note that only the mode I (hydrogen control) properties are strongly sensitive to grain shape. Grain size per se appears less important, judging from the similar SCC behavior of 7075 and 7090 with vastly different grain sizes and from the fact that commercial and HP7075 show similar RA losses after cathodic precharging. The larger differences between the two in SET tests is due more to slip planarity than to grain size variations, as will be discussed in the next section (11).

We recently completed a quite detailed study on HP7075 where we systematically varied the size and distribution of the PFZ and grain and grain boundary precipitates and then assessed the effects on hydrogen embrittlement in cathodically precharged and SET samples (12). The heat treatments produced three sizes of grain boundary n precipitates ranging from 20-70 nm, PFZ widths from 25-450 nm and different size distributions of

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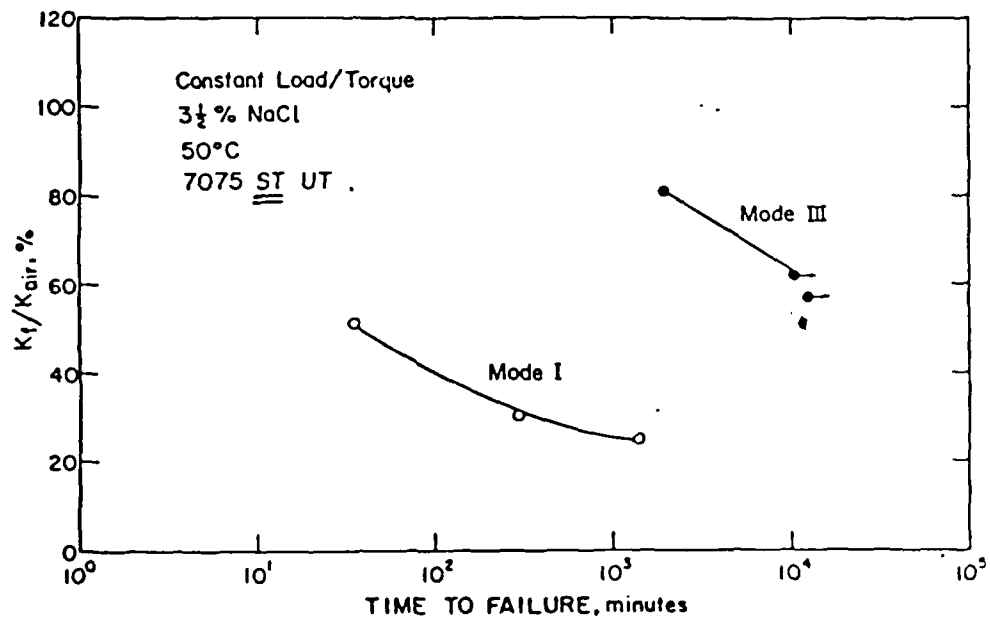
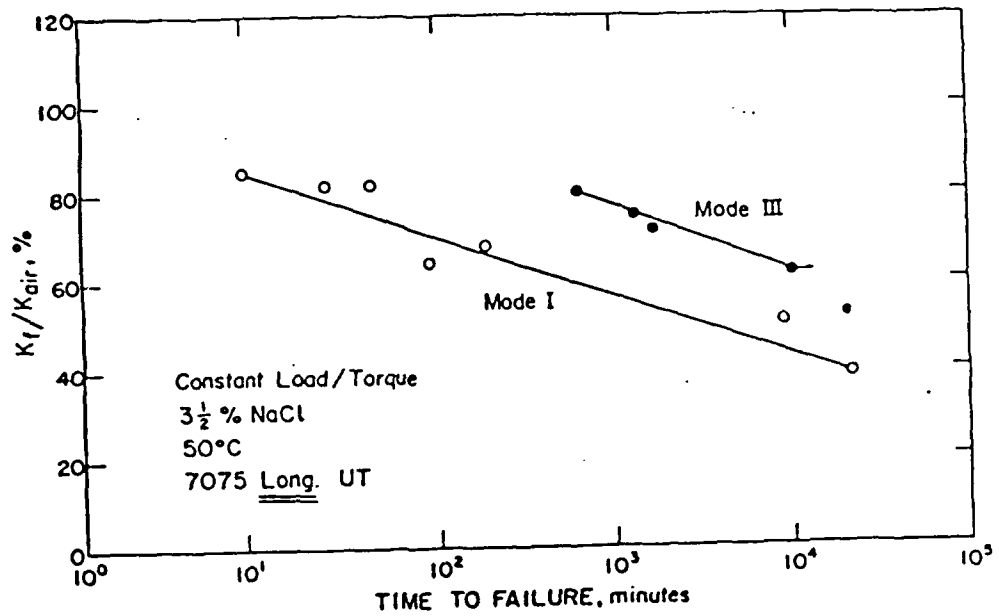


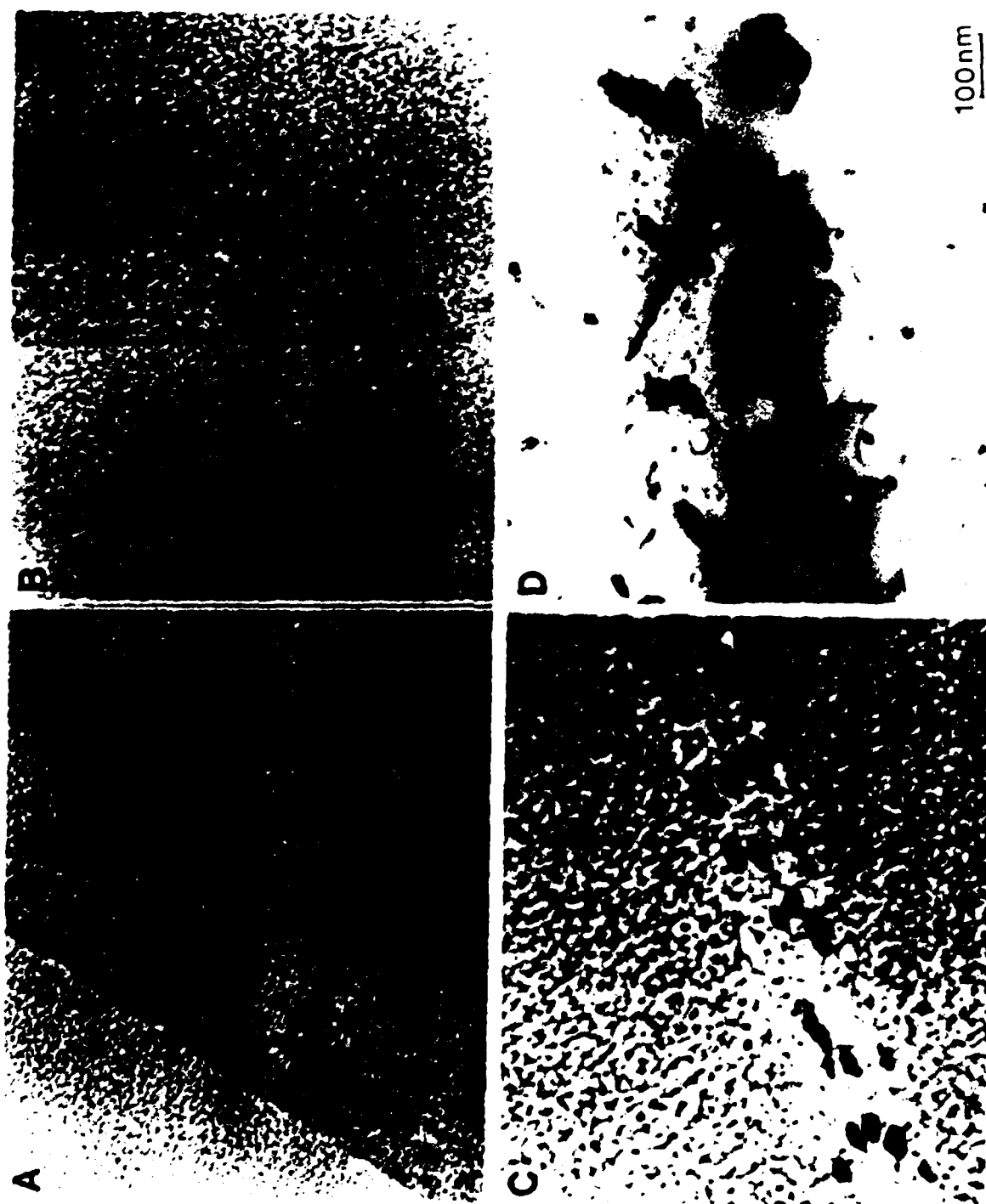
FIGURE 5. LOADING MODE RESULTS FOR 7075 UT IN LONG. AND ST DIRECTION

matrix GP zones  $\eta$  and  $\eta'$  precipitates. These are respectively illustrated in Figures 6-8, graphically illustrating the range of structures studied.

These tests revealed that different weights could be attributed to each of these features with regards to their contribution to hydrogen embrittlement. This is summarized in Figure 9, taken from a research highlight prepared for AFOSR, which emphasizes that the grain interior precipitates dominate susceptibility control, most likely through their ability to influence the degree of slip character, most importantly the degree of coarseness and/or planarity. We believe that these characteristics are quite general even when in commercial alloys, slip planarity is inhibited by the presence of a dispersoid population. Coarseness (spacing) differences are still possible, particularly on a local scale and precipitate control to encourage fine, homogeneous slip should become one of the important design tools for new alloys more resistant to environmental embrittlement. As anodic dissolution control becomes more important, the importance of homogeneous slip is not diminished, since we have shown in 7075 that slip band dissolution can be an important contributor to SCC(10).

The final issue to consider is the mechanistic implications of these observations and results. We have shown in a number of our studies that dislocation transport of hydrogen not only occurs in aluminum and its alloys, but that it is very difficult to rationalize observed behavior without this transport mechanism being the controlling parameter (11,13). Vivid support for this is seen in Figure 10, which demonstrates that in a SET test where large quantities of hydrogen can be transported by mobile dislocations, 100% intergranular hydrogen embrittlement occurs. In contrast, static precharging, which relies on lattice diffusion results in an intergranular zone only 1-2 grains deep from the surface. Clearly more extensive

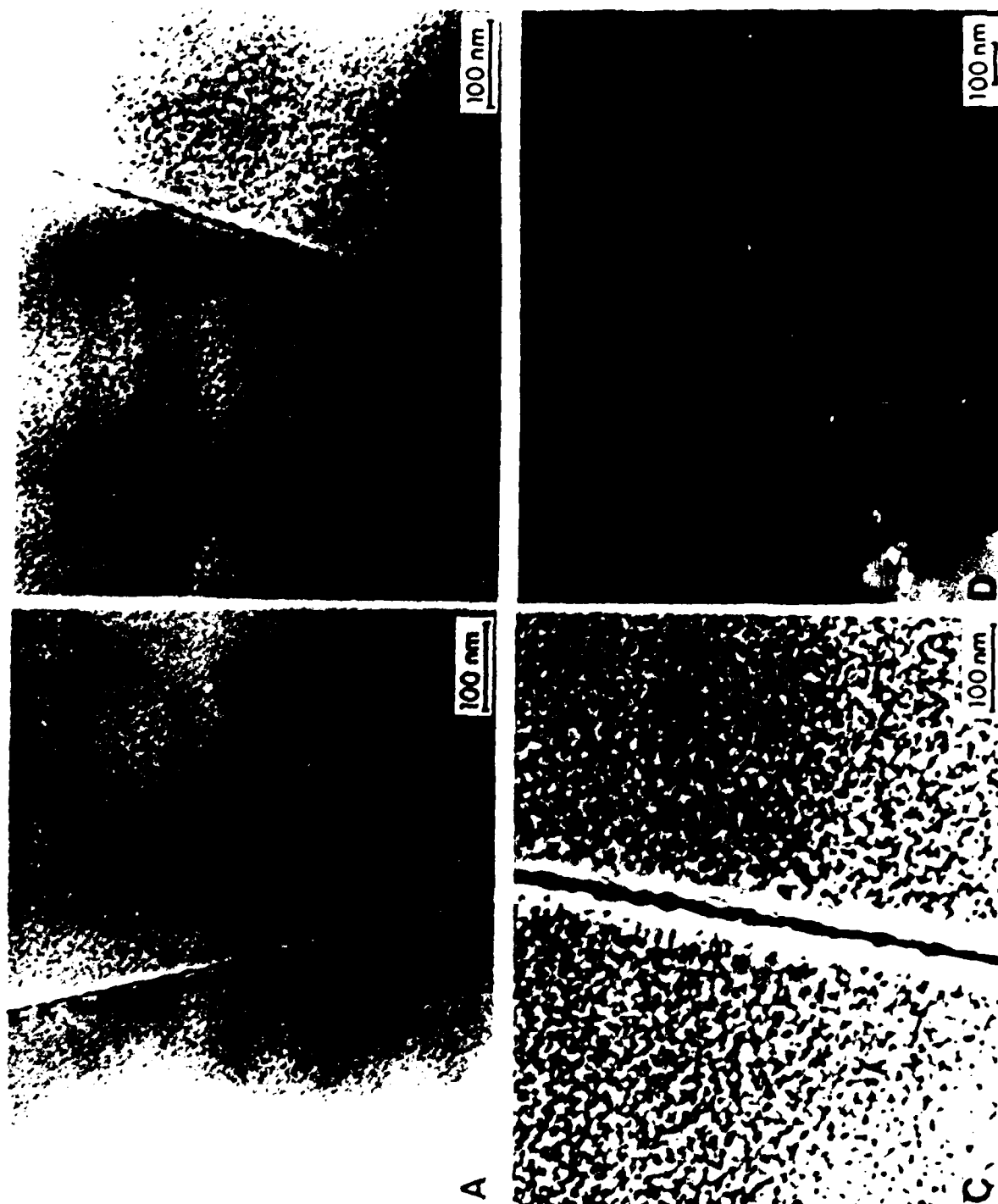
FIGURE 6. EFFECT OF HEAT TREATMENT ON GRAIN BOUNDARY PRECIPITATES  
IN HP7075



TYPICAL GRAIN BOUNDARY PRECIPITATES

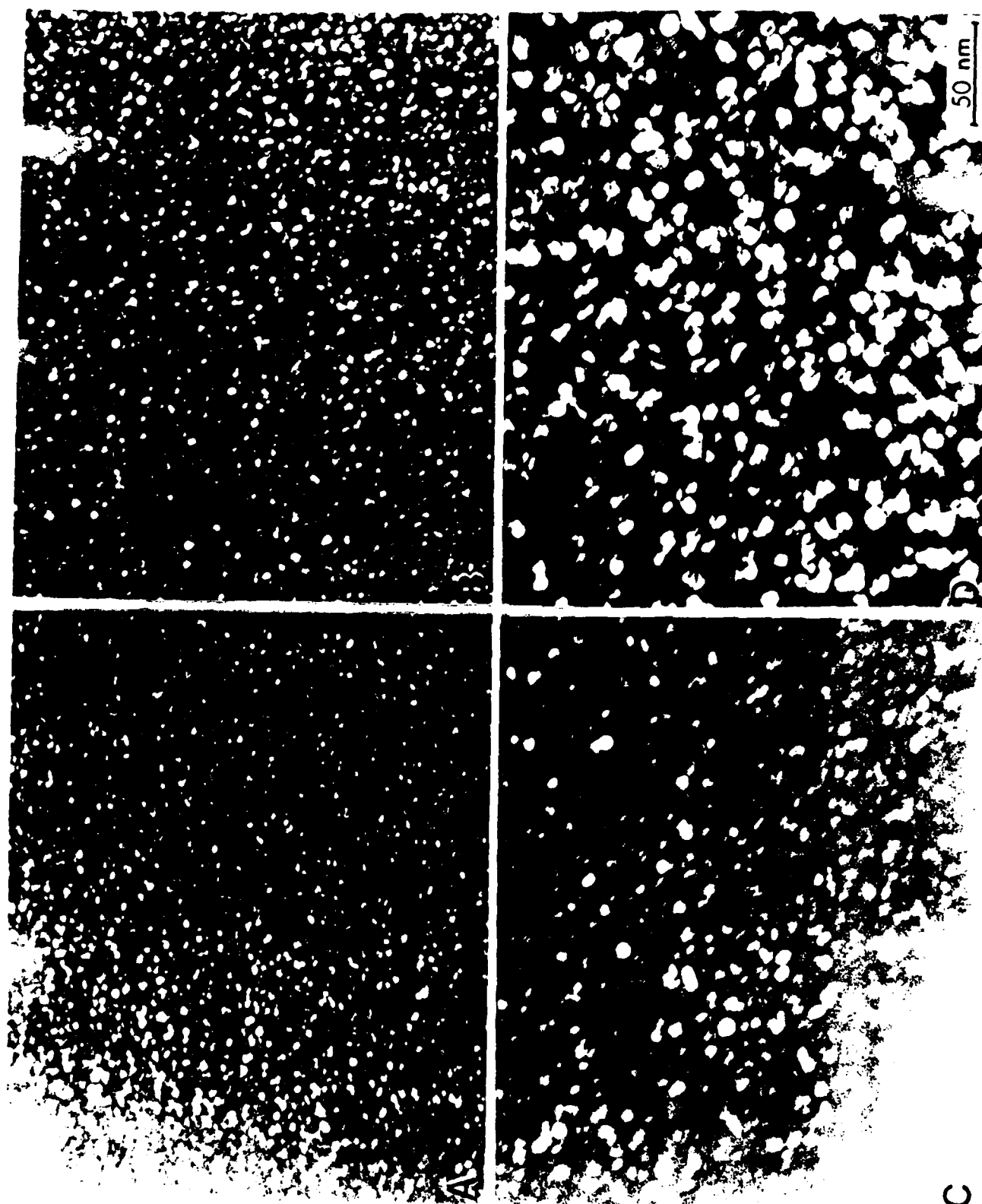


FIGURE 7. TYPICAL PRECIPITATION FREE ZONES AS A FUNCTION OF HEAT TREATMENT IN HP7075



TYPICAL PRECIPITATE FREE ZONE

FIGURE 8. DARK FIELD MICROGRAPHS OF MATRIX PRECIPITATES IN HP7075  
AS A FUNCTION OF HEAT TREATMENT



DARK FIELD MICROGRAPHS SHOWING  $\eta'$  AND  $\eta$  IN THE MATRIX

## MICROSTRUCTURAL FEATURES RESPONSIBLE FOR STRESS CORROSION CRACKING

### ● STRENGTHENING PRECIPITATES

7075 ( Al-Zn-Mg-Cu )

- Control 60% of susceptibility
- particles desirable for SCC resistance, essential for strength

### ● GRAIN BOUNDARY PRECIPITATES

- Control 30% of susceptibility
- precipitates desirable
- large size increases SCC resistance but lowers mechanical properties

### ● PRECIPITATE-FREE ZONE

- Controls <10% of susceptibility
- PFZ slightly desirable for SCC resistance, undesirable for strength

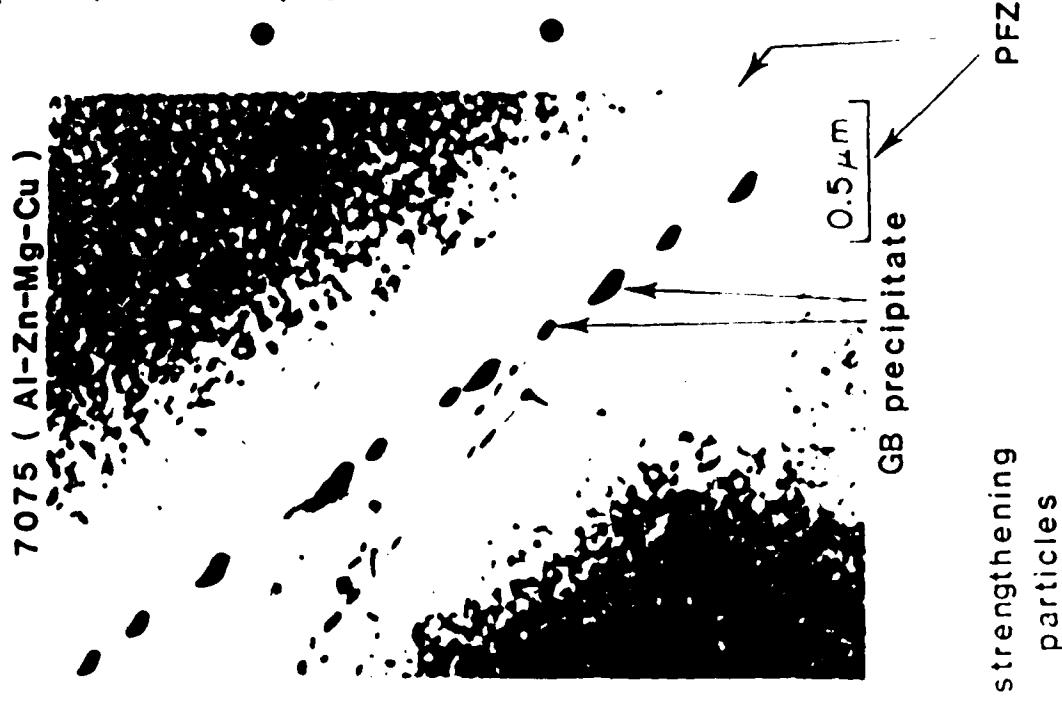


FIGURE 9. SUMMARY BEHAVIOR OF THE RELATIVE ROLES OF DIFFERENT MICROSTRUCTURAL UNITS ON STRESS CORROSION CRACKING OF HP7075

dislocation transport promoted by planar, coarse slip will exacerbate the occurrence of embrittlement; however this does not in of itself provide evidence of the mechanism by which it occurs.

Some of our recent work may provide some important clues. A study has been completed on the effect of cathodic charging temperature (20 or 150°C) on room temperature tensile behavior of HP7075(14). Embrittlement was observed after either treatment with the underaged temper most susceptible in both cases. However, intergranular fracture was only found following room temperature charging and evidence was presented to suggest that a grain boundary hydride, whose appearance is illustrated in Figure 11, catalyzed the onset of brittle fracture. Dislocation transport serves as the vehicle to transport the large quantities needed to transform to the hydride. The behavior following high temperature charging is different as the hydride may not be stable, but a different, as yet unknown mechanism takes over.

While still highly speculative the following description provides a self consistent picture of environmental embrittlement of high strength aluminum alloys at temperatures near ambient: Hydrogen generated from a local or more general cathodic reaction can penetrate short distances into the lattice or slightly deeper along grain boundaries, providing the oxide film is not an effective barrier. Plastic strain will both break the film and provide mobile dislocations generated from surface sources to act as dynamic transport paths. Hydrogen will be transported to local strong traps, with the depth of penetration controlled by the ability to retain coarse planar slip, most likely by sustained precipitate cutting. The hydrogen should then accumulate most strongly at large grain boundary precipitates, such as  $\eta$  and  $T$  particles. If the local fugacity becomes high

HP-7075 UT NOTCHED SET

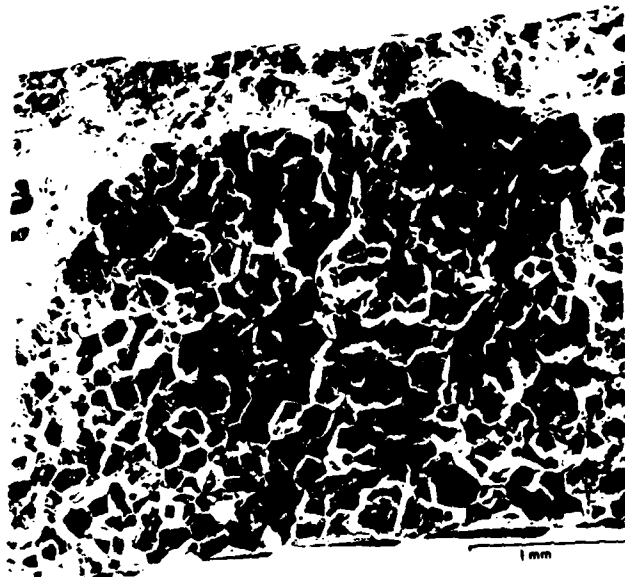


FIGURE 10. INTERGRANULAR FRACTURE OF HP7075 RESULTING FROM STRAINING ELECTRODE TESTING



FIGURE 11. HYDRIDE-LIKE FEATURES ON INTERGRANULAR SURFACE OF CATHODICALLY PRE-CHARGED HP7075-UT

enough, brittle hydride formation will promote the observed intergranular fracture. At lower fugacities particle cracking or decohesion will lead to crack initiation, with crack growth occurring as a result of hydrogen-induced softening or decohesion.

Concluding Remarks: We believe that the work summarized in this final scientific report has more than met the stated objectives of the program. Not only is the environmental embrittlement behavior of 7075 and 7090 now well detailed, but it is equally well understood. More importantly, the knowledge gained promises to have application in understanding the behavior of a much broader class of materials. We wish to sincerely thank the Air Force Office of Scientific Research and more specifically our technical monitor, Dr. Alan Rosenstein, for sustained support and encouragement during the period of this research.

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